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Efficacy of Surface Dust Treatments of Pirimiphos-Methyl and Etrimfos when Applied to Commercially Stored Wheat and Barley

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Abstract: Commercially stored wheat and barley were surface treated with dust formulations of pirimiphos-methyl and etrimfos at the manufacturer's recommended application rate. Samples were taken at four-weekly intervals for 32 and 16 weeks for wheat and barley respectively. Pesticide efficacy was determined using insect bioassays and chemical analysis of residues. There was no evidence to suggest a decline in residue levels of either pesticide over the experimental period. However, considerable variation was observed in the residue levels recorded at different sample points and also between residue levels recorded for the same point over the trial period. Control of susceptible strains of *Tribolium castaneum* (Herbst), *Oryzaephilus surinamensis* (L.) and *Sitophilus granarius* (L.) was achieved where recovered pesticide residues remained above 1 mg kg^{-1} pirimiphos-methyl and 1·6 mg kg^{-1} etrimfos.

Key words: grain, storage insects, pirimiphos-methyl, etrimfos, dust.

1 INTRODUCTION

There is a requirement for traded grain to be pest-free; EC regulation 689/92 states that 'cereals must be free from live pests (including mites) at every stage of their development'.[1] The most widely used method for the treatment and prevention of pest infestations is by the direct application of pesticides to the grain. This can involve applying an emulsifiable concentrate or dust formulation during conveyance. Cooling has been used as an alternative to chemical treatment. By reducing the temperature of the grain to 5°C, mite increase is delayed[2] and existing infestations of adult insects are killed.[3] However cooling still leaves the grain surface vulnerable to infestation and a surface dust treatment may be required.

The use of the more persistent pesticides, such as pirimiphos-methyl and etrimfos, has increased because of the need to store grain for longer periods and also for improved control of resistant populations.[4] As a consequence of this extensive use, concerns have grown regarding the possibility of pesticide residues appearing in cereal products such as flour and bread.[4] Wilkin and Fishwick[5] produced wholemeal flour and bread from wheat treated with organophosphorus pesticides, and found that approximately 50% of the pesticide present in the wholemeal flour survived the baking process.

The Maximum Residue Limits (MRLs) for organophosphates approved for use on stored grain have been reduced in the EU to 5 mg kg^{-1},[6] which is only slightly above the manufacturer's recommended application rate. However the need for a reduction in pesticide residues must be balanced by the need for effective pest control. Accurate information is therefore

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required as to which pesticides provide the most economic and effective protection. It is known from previous research that pesticides may degrade over prolonged storage periods[7] and that the biological activity may be reduced even though the pesticide is still present on the grain.[8] The loss of insecticidal activity has been attributed in part to the decrease in the availability of the pesticide to the insect.[9] The loss of biological efficacy may be explained by the gradual movement of the pesticide from the outer grain layers.[10]

Matthews and Maliphant[11] investigated the degradation of [^{14}C]pirimiphos-methyl on grain stored in the laboratory at 15°C for 28 weeks. Breakdown was found to be slow, with 77% of the applied dose remaining unchanged by the end of the experiment. The effectiveness of unlabelled pirimiphos-methyl was assessed against organophosphorus-resistant *Oryzaephilus surinamensis* (L.) and *Tribolium castaneum* (Herbst). One hundred percent mortality of *O. surinamensis* was maintained during the 28 weeks, with some survival of *T. castaneum* occurring after 12 weeks storage. The decrease in mortality of *T. castaneum* could not be correlated with the loss of pirimiphos-methyl from the grain to which it was exposed nor with any decrease in the extractable pirimiphos-methyl observed in the radiotracer experiment. It was suggested that the insecticide had become less available to the insects by being relocated more deeply within grain tissues.

Thomas *et al.*[12] investigated the decline of residues of chlorpyrifos-methyl, pirimiphos-methyl, fenitrothion and etrimfos on wheat on an experimental scale (20 tonne) under ambient conditions in the UK, using insect bioassay tests. Each of the four pesticides produced >95% mortality of all insect species tested for 36 weeks, with the exception of fenitrothion, where survival of susceptible *T. castaneum* increased after 32 weeks. Comparatively little decay of any of the pesticides occurred over the storage period.

Le Patourel[13] investigated the variation in residue levels of pirimiphos-methyl and etrimfos in wheat and barley treated at the recommended application rates under commercial conditions, using standard application equipment. There was no evidence for decline in pesticide residues over the trial period of six to eight months. Complete kill of susceptible *O. surinamensis* was achieved at the end of the trial for both wheat and barley treated with either pesticide.

Wilkin and Stables[14] investigated the effects of pirimiphos-methyl, etrimfos and methacrifos dusts on mites in the surface layers of commercially stored barley. Etrimfos was found to be the most effective compound in the field and laboratory. However, there is no information currently available regarding the efficacy of surface dust treatments, against insect pests.

The type of commodity that is treated may also affect the biological efficacy of the pesticide. Al-Saffar and Al-Iraqi[15] found that the effectiveness of pirimiphos-methyl and malathion against *Trogoderma granarium* (Everts) and *Tribolium confusum* (Jacqueline Du Val) was greater on wheat than on maize, and greater on maize than on barley for a given rate.

This trial therefore aimed to investigate the decline of pirimiphos-methyl and etrimfos residues when applied as a dust surface treatment to commercially stored wheat and barley. The efficacies of different pesticides when applied to different commodities were compared using insect bioassay techniques and chemical analysis of pesticide residues.

2 MATERIALS AND METHODS

2.1 Field site

Two bays at a grain store in Lincolnshire, England were used in this trial. Each bay measured 30 × 18 m and had a capacity of approximately 3250 tonnes. The untreated grain had been in store for about three months prior to treatment. One bay contained spring wheat and the other spring barley from the 1992 harvest. Each bay was cooled by a suction aeration system controlled manually by store staff.

2.2 Insects

The insects used for the bioassay assessments were laboratory susceptible strains of *Tribolium castaneum* (Herbst), *Oryzaephilus surinamensis* (L.) and *Sitophilus granarius* (L.). All had been reared at the Central Science Laboratory (CSL) in constant conditions of 25°C and 70% RH. Adults of known age were used for the test; *S. granarius* when two to four weeks old, *O. surinamensis* when zero to two weeks old and *T. castaneum* when three to five weeks old.

2.3 Treatment of grain

Dust formulations containing 20 g kg^{-1} pirimiphos-methyl ('Actellic') and etrimfos ('Satisfar') were used for the trial. Treatments were carried out in November 1992. Each bay was divided into three equal areas measuring 10 × 18 m. One area was designated to be treated with pirimiphos-methyl, one with etrimfos and the third remained untreated to act as a control. Each area was marked out into 1 × 18 m strips with bamboo canes.

The pesticides were applied to the grain surface using a method described by Wilkin and Stables.[14] The quantity of dust required to give a dose of 4 mg kg^{-1} in each strip was calculated as being equivalent to 50 g per

square metre of surface area, assuming a treated depth of 0·3 m. The amount of dust was weighed out and sprinkled evenly by hand from a plastic bag onto the grain surface. The grain was then raked using a wooden rake, 1 m wide with 10 cm tines. When all the strips had been treated the canes were removed. Five sample points were then marked in each area by canes placed equidistant from each other, down the centre of the area, across the width of the bay. The sample points were designated C1–C5 in the untreated wheat and C6–C10 in the untreated barley. Sample points PM1–PM5 were in the wheat treated with pirimiphos-methyl, and PM6–PM10 in the barley treated with pirimiphos-methyl. Similarly sample points E1–E5 and E6–E10 were in the wheat and barley respectively, treated with etrimfos.

2.4 Sampling of the grain

At week 0 (immediately following treatment) and then at four-weekly intervals, samples of grain (approximately 700 g) were removed from the top 10 cm at each sample point in each area, using a metal scoop. These were put into plastic bags, transferred back to the CSL and stored for one day prior to use in insect bioassays and pesticide residue analysis. The samples were collected for 16 weeks in the case of the barley, 28 weeks for points PM2 and PM3 and 32 weeks for the other wheat sample points. The collections terminated at these times because the grain was sold.

2.5 Assessment of physical conditions of grain

Measurements of moisture content were taken from five sample points in each bay using an electrical resistance meter.

The grain temperatures at the points were taken using a 'Pnemac' hand-held temperature probe. The probe was gently pushed into the grain to a depth of 1 m and left to equilibrate for approximately two minutes before the readings were taken. The ambient temperature was recorded every 60 min using a 'Squirrel' data logger and the weekly means were calculated using a 'Grant Squirrel' software analysis package. However, due to circumstances beyond our control, it was not possible to obtain complete temperature records. Grain temperatures could not be taken before week 19, and as the barley had been sold by week 19, only four readings could be taken in the wheat from week 19 to week 32. Also the ambient temperatures could only be taken up to week 19.

2.6 Insect bioassay

The samples of grain collected for bioassay were each divided into batches (50 g) and put into wide-necked jars (120 ml). Four replicates were prepared for each sample point per test species. The jars were closed with a filter paper lid and left to equilibrate overnight at the test conditions of 25°C and 70% RH. After this period batches of 25 insects were removed from culture and placed into each jar. The jars were then closed with a filter paper lid and kept in the test conditions for seven days. The contents of each jar were then emptied onto an enamelled tray and the numbers of live, knocked-down and dead insects were recorded. An insect was considered knocked down if it was on its back and unable to right itself, even when aided with a small brush. It was considered dead if no movement was observed.

2.7 Pesticide residue analysis

At each assessment period samples of wheat and barley from each sampling point were sent to be analysed for pesticide residues using the Panel method.[16]

3 RESULTS

3.1 Pesticide residue analysis

Table 1 shows the means and standard deviations for the pesticide residues in each commodity at each sampling point over the entire experimental period. The data were subjected to analysis of variance, and showed no significant difference ($P > 0{\cdot}05$), between the means of the residues (taken over all the sampling points) at different sampling times, indicating no evidence for decline in residue levels of either pesticide on either wheat or barley. There was, however, a significant difference ($0{\cdot}01 < P < 0{\cdot}05$) in the mean residues (taken over all the sampling times) at different sampling points

TABLE 1

Pesticide Residues Detected in Each Commodity at Each Sampling Point Over the Entire Experimental Period

Commodity/ Sample point	*Pesticide residues (mg kg^{-1}) ($\pm SD$)* — *Pirimiphos-methyl*	*Etrimfos*
Wheat		
1	7·5 (±4·9)	5·6 (±3·2)
2	4·3 (±4·5)	0·9 (±0·4)
3	5·9 (±3·2)	1·9 (±1·2)
4	3·4 (±1·7)	7·1 (±5·1)
5	2·8 (±1·5)	6·1 (±3·4)
Barley		
6	4·8 (±4·0)	2·5 (±0·9)
7	4·5 (±3·0)	1·9 (±1·0)
8	2·0 (±2·1)	4·0 (±2·1)
9	5·3 (±2·7)	1·8 (±1·4)
10	4·7 (±2·8)	3·7 (±1·0)

for both pesticides on wheat and for etrimfos on barley, indicating a variation in treatments at different sample points in these areas. There was no significant difference ($P > 0.05$) in the residues at the different sampling points in the barley treated with pirimiphos-methyl.

3.2 Insect bioassay

High control mortalities (>5%) were observed at week 0 with all insect species exposed on untreated wheat and with *O. surinamensis* and *S. granarius* on untreated barley. At this period maximum mean control mortalities of 11%, 15% and 100% occurred with *S. granarius*, *T. castaneum* and *O. surinamensis* respectively at sample point C1 of the untreated wheat. Results of the residue analysis indicate that at this assessment period 0·2 mg kg^{-1} etrimfos and 0·1 mg kg^{-1} pirimiphos-methyl were present on the wheat. On barley at week 0, a maximum mean control mortality of 6% was achieved with *O. surinamensis* at sample points C7 and C10, and 9·1% mortality occurred with *S. granarius* at sample point C9. Results of the residue analysis show that at these sample points approximately 0·1 mg kg^{-1} etrimfos and pirimiphos-methyl were present on the barley.

At all other assessment periods, control mortality did not exceed 3% with *T. castaneum* on both commodities. With *O. surinamensis* however, control mortalities ranged from 0 to 16% on wheat and 0 to 13% on barley. Similarly, control mortalities for *S. granarius* ranged from 0 to 15% on wheat and 3 to 13% on barley. Where the high mean control mortalities were recorded, the numbers of dead insects in individual replicates were found not to vary greatly, indicating that the high means were not due to one replicate having an unusually high number of dead insects. Also the sample points and assessment periods where the high mean control mortalities occurred, varied with each species, indicating that the high mortalities did not occur at specific points or times. The results of residue analysis showed low levels of pesticide residues (<0·08 mg kg^{-1}) at all sample points and assessment periods.

The majority of the pesticide treatments produced mean mortalities of 100% up to the final assessment period. However, at certain sample points and assessment periods complete mortality was not achieved. When exposed to the wheat treated with pirimiphos-methyl from point PM2 at week 12, low mortalities of 19% and 14% were recorded for *T. castaneum* and *S. granarius* respectively, and 94% mortality occurred with *O. surinamensis*. Also at weeks 16 and 19, 98% and 95% mortalities of *T. castaneum* and 83% and 81% of *S. granarius* were recorded for the respective weeks.

With the barley treated with pirimiphos-methyl from point PM8, mortalities of only 6% and 13% of *T. castaneum* and *S. granarius* respectively were recorded at week 9; and at week 12, mortalities of 17%, 2% and 20% were recorded for *T. castaneum*, *S. granarius* and *O. surinamensis* respectively. With barley from PM9, 90% mortality of *S. granarius* was observed at week 9, and point PM10 produced 94% mortality of *T. castaneum* at week 4.

When exposed to wheat treated with etrimfos from point E2, mean mortalities of 10% and 64% of *T. castaneum* and *S. granarius* respectively were recorded at week 9. At week 16, mortalities of 0%, 10% and 41% were recorded for *T. castaneum*, *S. granarius* and *O. surinamensis* respectively. From weeks 19 to 32, the mortality response of *T. castaneum* decreased from 82% to 15%. With *S. granarius*, mortalities ranged from 10% at week 19, to 69% at week 28; however, this decreased to 2% at week 32.

With wheat taken from E3, mortalities of 90%, 91%, 81% and 58% of *T. castaneum* were recorded at weeks 9, 16, 19 and 28 respectively. At week 32, 0%, 6% and 94% mortalities of *T. castaneum*, *S. granarius* and *O. surinamensis* respectively were recorded. A mean mortality of 90% of *T. castaneum* was also recorded at week 24 when exposed to wheat from E4.

In tests with etrimfos on barley, a mortality of 20% of *T. castaneum* was recorded at week 4 on grain taken from E6. When exposed to barley from E7, mortalities of 6% and 67% of *T. castaneum* and *S. granarius* respectively occurred at week 0, with 48% and 71% mortalities of *T. castaneum* recorded at weeks 4 and 12 respectively. With the barley from E9, 76% mortality of *T. castaneum* was recorded at week 0, with 29% and 12% mortalities of *T. castaneum* and *S. granarius* respectively recorded at week 16.

Comparing the results of the pesticide residue analysis and the insect bioassays shows that, in the majority of cases, >95% mortality of *T. castaneum* occurred at doses of above about 1 mg kg^{-1} and 1·6 mg kg^{-1} for pirimiphos-methyl and etrimfos respectively when applied to either wheat or barley. For *O. surinamensis*, doses of more than 0·4 mg kg^{-1} pirimiphos-methyl and 0·5 mg kg^{-1} etrimfos resulted in increased mortality on both commodities. For *S. granarius*, mortalities of >95% were observed at doses above 0·9 mg kg^{-1} and 1·4 mg kg^{-1} pirimiphos-methyl and etrimfos respectively when applied to either wheat or barley.

Where residue levels were lower, a reduction in mortality was observed. Specific examples of this were observed at PM2 on week 12, where the level of pesticide residue was recorded as 0·4 mg kg^{-1}, which resulted in 19% mortality of *T. castaneum*. Similarly at E7 on week, 0, 67% mortality of *S. granarius* occurred where a pesticide residue of 0·7 mg kg^{-1} was recorded. In most cases where the residue levels remained above these levels, complete control of the insects was achieved for the entire experimental period. For

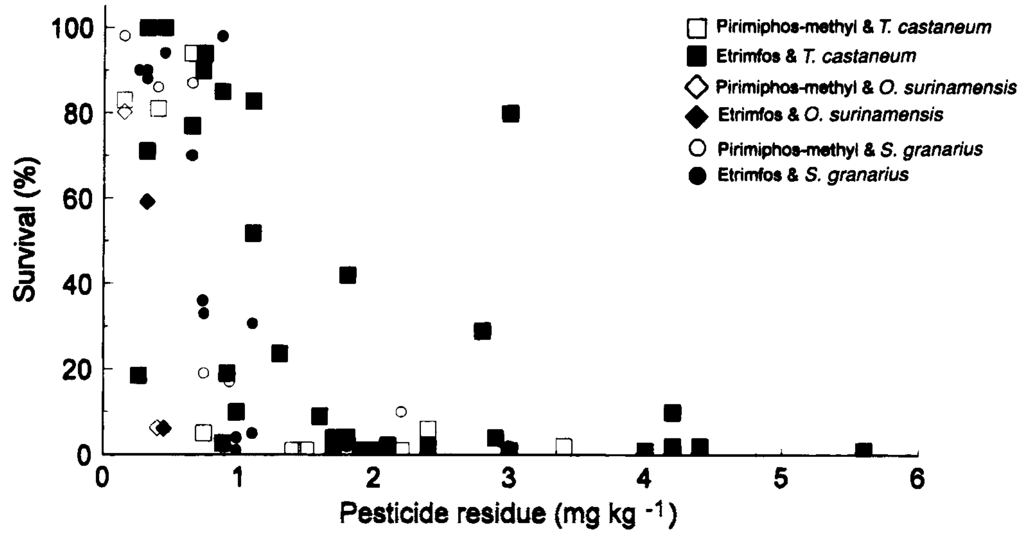

Fig. 1. Scatter diagram of pesticide residue versus survival.

instance, at sample point E2, etrimfos residue levels did not exceed 1·6 mg kg^{-1} for the entire experimental period. With *T. castaneum*, 100% mortalities were only recorded where residue levels were 1·5 mg kg^{-1} and 1·6 mg kg^{-1}.

There were, however, a few anomalies where a dose of above 1·6 mg kg^{-1} did not produce >95% mortality. For example, at E6 at week 4, a pesticide residue level of 3 mg kg^{-1} resulted in only 20% mortality of *T. castaneum*. Also at E7 at week 12, a residue level of 2·8 mg kg^{-1} resulted in 71% mortality of *T. castaneum*. Conversely at E9 at week 9, a residue level of 1 mg kg^{-1} resulted in 96% mortality of *S. granarius* and 100% mortality of *T. castaneum*.

Figure 1 is a scatter diagram showing graphically the mean percentage of insect survivors at individual sample points and assessment periods, against the recovered pesticide residue. It shows that the majority of survivors were observed at doses lower than approximately 1·5 mg kg^{-1}.

3.3 Physical assessments

There was a significant difference ($P < 0{\cdot}05$) between the mean moisture content measurements (taken over all the sampling points) at different times (Table 2), indicating a fluctuation in this parameter over the experimental period. The highest measurements were recorded during the months of February and March.

The mean temperature recordings of the wheat taken at weeks 19 to 32, were 4·7(±0·4), 7·2(±0·2), 9·3(±0·5) and 12·2(±1·3)°C respectively. The highest temperature of 14·5°C was recorded at the end of the experimental period in June. The weekly mean ambient temperatures for weeks 0, 4, 9, 12, 16 and 19 were recorded as 9·6, 6, 5·6, 5·6, 5·2 and 9·6°C respectively.

4 DISCUSSION

The aim of this experiment was to assess the efficacy of surface dust treatments of pirimiphos-methyl and etrimfos on wheat and barley under practical storage conditions, using insect bioassay techniques together with chemical analysis of residues. The effectiveness of each pesticide on the two commodities, however, could not be directly compared because the residue levels were so variable at each sample point. The information

TABLE 2
Moisture Content of Samples taken from Five Sample Points in the Wheat and Barley Bays

	Moisture content (% m/m) (±SD)								
	Time after treatment (weeks)								
Commodity	*0*	*4*	*9*	*12*	*16*	*19*	*24*	*28*	*32*
Wheat	15·36 (±0·39)	15·82 (±0·36)	16·54 (±0·72)	17·42 (±0·59)	17·42 (±0·81)	16·74 (±0·15)	16·02 (±0·29)	15·28 (±0·34)	14·4 (±0·3)
Barley	15·88 (±1·1)	15·14 (±0·94)	16·88 (±0·95)	17·44 (±0·92)	17·16 (±0·82)	—	—	—	—

that can be gained from the results, indicates that, in the majority of cases, doses above 1 mg kg^{-1} pirimiphos-methyl and 1·6 mg kg^{-1} etrimfos provided >95% mortality of *T. castaneum;* doses above 0·4 mg kg^{-1} pirimiphos-methyl and 0·5 mg kg^{-1} etrimfos resulted in >95% mortality of *O. surinamensis* and doses above 0·9 mg kg^{-1} pirimiphos-methyl and 1·4 mg kg^{-1} etrimfos produced >95% mortality of *S. granarius.*

Thomas *et al.*[12] found that treatments with pirimiphos-methyl and etrimfos produced 100% mortalities of susceptible strains of *O. surinamensis, T. castaneum, S. granarius* and *Sitophilus oryzae* (L.) for 36 weeks. The pesticide residues of pirimiphos-methyl remained constant at about 3·5 mg kg^{-1} for the entire experimental period. With etrimfos there was an initial drop in residues from 5 mg kg^{-1} to 4·2 mg kg^{-1} which then remained stable for the remainder of the trial.

Le Patourel[13] found that complete control of a susceptible strain of *O. surinamensis* was achieved on barley, where pirimiphos-methyl residues averaged 2·94–5·27 mg kg^{-1} and etrimfos residues averaged 2·37–5·95 mg kg^{-1}; and on wheat where pirimiphos-methyl and etrimfos residues averaged 0·73–1·9 mg kg^{-1} and 0·99–2·21 mg kg^{-1} respectively, for six to eight months.

The anomalies observed in this investigation where a dose above 1·6 mg kg^{-1} etrimfos failed to provide complete kill of insects cannot be fully explained, but may be attributed to the movement of some of the insecticide from the surface to the interior of the grain making it unavailable to the insects during a short term bioassay.[9,10]

It has been observed (Binns, T. J. & Henderson, S., unpublished results) that 99·9% mortalities of susceptible strains of *T. castaneum, O. surinamensis* and *S. granarius* were obtained at doses of 1·1 mg kg^{-1}, 0·18 mg kg^{-1} and 0·42 mg kg^{-1} pirimiphos-methyl; and doses of 0·57 mg kg^{-1}, 0·21 mg kg^{-1} and 0·61 mg kg^{-1} etrimfos respectively. The results of this work and the present research indicate that, if an intended recommended dose of 4 mg kg^{-1} could be accurately applied to the grain, complete control of susceptible strains of insects would be achieved. However it is known that, in practice, accurate application of the intended dose is difficult to achieve and 50% or more of the dose can be lost during application under practical conditions.[17]

The insects used in this experiment were laboratory susceptible strains. In the field situation, however, resistant strains may be encountered and a higher dose required to provide complete protection. Binns and Henderson[18] reported an $LD_{99.9\%}$ of approximately 0·5 mg kg^{-1} pirimiphos-methyl and 0·2 mg kg^{-1} etrimfos against field strains of *O. surinamensis.* Le Patourel[13] also found that wheat samples treated with pirimiphos-methyl and etrimfos failed to control field strains of *O. surinamensis* at doses of less than 1·7 mg kg^{-1} pirimiphos-methyl and 2·1 mg kg^{-1} etrimfos.

Although the insect bioassay is a good indicator of pesticide efficacy, it should not be taken as an absolute measure of effectiveness because the assessments were made at 25°C and 70% RH. The effectiveness of pesticides has been shown to be related to temperature. Tyler and Binns[19] and Barson[20] have shown that, when applied to filter papers, insecticide effectiveness is greater at 25°C than at lower temperatures. Organophosphorus pesticides also show a positive temperature coefficient, i.e. toxicity is greater when the insects are held at higher temperatures after dosing.[21] Therefore the level of mortality observed in the bioassays may be greater than would occur under field conditions where the temperature might be lower.[12] However under field conditions the insects might also be exposed to the pesticide for longer than the seven-day exposure period of the bioassay, and this effect could be reversed.[12]

At low temperatures the rate of chemical degradation is slower;[21] therefore, although insecticide effectiveness is reduced, persistence is increased. In this experiment no evidence for decline in residue concentrations was observed during the trial period, which may be attributed to the low grain and ambient temperatures recorded.

The relatively high control mortalities (>5%) observed at week 0 may be explained by contamination of the grain just after treatment. This was confirmed by the results of the residue analysis. It was observed that, during the treatment, a number of pesticide dust particles were present in the air, and these may have become deposited on the control area. However, after week 0, low pesticide residues (<0·05 mg kg^{-1}) were recorded in the control area. The high mortalities observed with *O. surinamensis* after week 0 may be expected because of the long exposure period on whole grains. However, the unusually high control mortalities of *S. granarius* after seven days' exposure were unexpected and the cause remains unclear.

The results of the pesticide residue analysis from each sample point indicate the large range of residue levels obtained. This highlights the difficulties in applying the recommended application rate evenly over the grain surface. An added problem in this case was that the grain sloped quite considerably from the centre to the sides of the store. Consequently sample points at the bottom of the slope may have received a higher dose, due to movement of treated grain down the slope. This can be seen at points PM1 and PM6, where very high residue levels were recorded. Disturbance of the grain also occurred during sampling at each assessment period which may have contributed to the uneven distribution.

By the end of the experimental period some sample points were recorded as having residues above the

MRL, e.g. at PM1 and E1, 15·5 mg kg^{-1} pirimiphos-methyl and 8·2 mg kg^{-1} etrimfos respectively were recorded. These high levels would not have presented a safety hazard however, since when the grain was moved for sale, the mixing would have resulted in negligible overall residue levels.

5 CONCLUSIONS

This trial has shown that, in general, >95% mortality of susceptible strains of *T. castaneum*, *O. surinamensis* and *S. granarius* was achieved on both commodities for the entire experimental period, when exposed to recovered pesticide residues of at least 1 mg kg^{-1} pirimiphos-methyl or 1.6 mg kg^{-1} etrimfos.

No evidence of decline in pesticide residues was observed during the experimental period; however, there were considerable variations in the residues detected at different sample points.

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